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Section 8d

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Dear Sir or Madam:

As required by 40 CFR 716, as amended, we herewith submit a copy of the following recently completed health and safety study.

Fate of MDI in Water Part I

Chemical Name	CAS Number
MDI monomer 2,4' isomer	05873-54-1
MDI monomer 4,4' isomer	00101-68-8
MDI monomer 2,2' isomer	02563-05-2
MDI isomer Mixture	26447-40-5
Polymeric MDI	09016-87-9
4,4' MDA	00101-77-9

The III report identification number 10807, has been marked on the title page of the report. Please refer to this identification number in any communication regarding this study. **The enclosed report does not contain any Confidential Business Information.**

The study is sponsored by the International Isocyanate Institute on behalf of the following:

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Very truly yours,

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FE-E-74

10807

Study on the fate of MDI in water

FE-E-74 Part 1

Studies on

Experimental method for vigorous stirring experiment

and

Analytical method of MDI in water

November 27,1990

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Contents

Synopsis

1. Introduction

2. Study on experimental method for vigorous stirring experiment of MDI

  2-1 Experiments

    (1) Materials

    (2) Preparation of the dispersed solution of MDI

    (3) Vigorous stirring experiment based on the analysis of individually prepared solution

  2-2 Results

    (1) Preparation of dispersed solution using dispersing agent

    (2) Vigorous stirring experiment based on the analysis of individually prepared solution

    (3) Detection of reaction product

3. Study on analytical method of MDI in water

  3-1 Experiments

    (1) Materials

    (2) Reaction conditions of the coupling with DBA: reaction time and the amount of DBA added

    (3) Analytical method of MDI in water

    (4) Influence of MDA on analysis of MDI in water

    (5) HPLC condition

  3-2 Results

    (1) Reaction conditions of the coupling with DBA

    (2) Analytical method of MDI in water

    (3) Influence of MDA on analysis of MDI in water

    (4) Recommended condition of the analysis of MDI using DBA

4. Discussion

Synopsis

As a preparatory experiment of the study on the fate of diphenylmethanediisocyanate (MDI) in water, the experimental method suitable for vigorous stirring experiment of MDI and the analytical method of MDI in water which is not influenced by the existence of diphenylmethanediamine(MDA) were investigated and the following results were obtainded.

(1)The preparation of the dispersed solution of MDI was tried using HCO-20 and olive oil as dispersing agents, but MDI reacted with the dispersing agents.

(2) MDI was added to 20 ml of water at nominal concentration of 1000 μl/l and stirred with magnetic stirrer. The time course of the disappearance of MDI in water was measured by analyzing the residual amounts of MDI in individually prepared solutions during the disappearance.

Under this condition, the stop of stirring during the experiment due to the adhesion of stirrer bar to glass vessel did not occured and the smooth disappearance curve of MDI was obtained. Accordingly the method is able to be employed as one for vigorous stirring experiment of MDI.

The disappearance rate was accelerated by a factor of about two in half-life period when glass beads were added in test solution probably as results of the enlargement of adhesion area of MDI.

(3) MDI was analyzed by coupling with dibutylamine (DBA), followed by quantification with HPLC on ODS column. The good relationship between peak area and the amount of MDI and the good separation of two isomers of MDI were observed for this anlytical method.

The extraction of MDI in water and the deactivation of isocyanate group were conducted simultaneously by extracting MDI in water with toluene solution of DBA

The conditions to achieve high recovery of MDI in water was determined.

This method was not influenced by the existence of MDA in water and consequently it is able to be employed as the quatification method of the residual MDI in study of the fate of MDI in water.

(4) The fate of MDI in water was evaluated preliminarily.

1 Introduction

Since isocyanate reacts easily with water to produce the corresponding amine, polyurea, etc. because of the reactive nature of isocyanate group, it is presumed that the effect of the spilled isocyanate on the aquatic environment is not caused by isocyanate itself, but by its reaction product in water.

From this point of view, in FE-E-63 project the fate of tolylene diisocyanates (TDI) in water were examined under two typical conditions, static and vigorous stirring conditions, prior to the toxic test to aquatic organisms.

When TDI was stirred vigorously in water, it disappeared within 24 hours to produce TDA, oligourea and polyurea, in which about 5 percent of total reaction products was water-soluble including TDA, mono-urea and di-urea. On the other hand, under static condition the disappearance rate of TDI was largely slow compared with one under vigorous stirring condition. The disappearance rate of TDI in water was also dependent on the composition of isomer in TDI under both conditions. Toxicity tests (FE-E-66 project) were conducted with Oryzia latipes, Daphnia magna and Chlorella vulgaris using test solutions prepared by both conditions and it was found that the raction product of T-80 in water was the most toxic to daphnia and much less toxic to fish and alga judging from toxicity values based on nominal concentrations. It was also inferred that some toxic reaction products other than TDA, such as T-80 itself and oligourea, exist in the reaction mixture.

Diphenylmethanediisocyanate (MDI) is another largely used raw material of polyurethane and it is significant to assess the effect of MDI on the aquatic environment. FE-E-74 project aim to elucidate the fate of MDI in water prior to toxicity test of it.

As the first step of the study on the fate of MDI in water, preliminary experiment was conducted to examine the applicability of the experimental methods used in TDI experiment to MDI and it was found that there are following two technical problems to be solved before conducting the study on the fate of MDI in water. One is the experimental method suitable for vigorous stirring experiment of MDI:

Since TDI was dispersed homogeneously in water, time course of disappearance of TDI was followed by analyzing the residual amount of TDI in the aliquot taken out from common reaction solution.

When MDI, however, was stirred with magnetic stirrer under the same condition as TDI experiment, MDI was not dispersed in water but the majority of MDI were adhered to Teflon stirrer bar and the inner wall of glass vessel in a lump and occasionally the stirring was stopped because of adhesion of stirrer bar to glass vessel through MDI adhered on Teflon stirrer bar. These results indicate that the experimental method used in TDI experiment can not be applicable to MDI and it is necessary to establish the experimental method suitable to MDI.

Another problem is the lack of suitable analytical method of MDI in water: in FE-E-63 project, TDI in water was extracted with toluene and reacted with pyridyl piperidine, followed by quantification with HPLC. This method, however, is not able to be employed for MDI because MDA, a possible reaction product of MDI in water, is coextracted with toluene and coupled with the residual MDI in toluene before next reaction with pyridyl piperidine, which lower apparently the quantified amount of residual MDI. Accordingly new analytical method of MDI in water which is not influenced by MDA is required in study on the fate of MDI in water.

The purpose of present investigation (FE-E-74 part 1) is to solve these problems and to establish the experimental methods necessary to study the fate of MDI in water.

In this study the mixture of 2,4' - and 4,4' - monomeric MDI was used as test substance since it is thought to be easy to operate it because of liquid, though the final object of FE-E-74 project is to elucidate the fate of polymeric MDI in water.

In this report two problems were described separately.

2. Study on experimental method for vigorous stirring experiment of MDI

Two methods were examined as method of vigorous stirring experiment of MDI.

One is the method using the dispersed solution of MDI in water prepared using the appropriate dispersing agent. If the preparation of the dispersed solution is possible, the same method as TDI is able to be used and then the preparation of the dispersed solution was examined using dispersing agent.
Another method is to analyze the residual amount of MDI in individually prepared solutions at each time of the time course of the disappearance of MDI. Since in this method most of MDI added are adhered to Teflon stirrer bar as observed in preliminary experiment, there is the possibility that the stirring stops during the experiment because of the adhesion of stirrer bar to glass vessel as observed in the preliminary experiment and that the reproducibility of kinetic data of disappearance becomes poor because of poor contact of MDI with water. However, if there are the adhesion site other than Teflon stirrer bar or if the amount of MDI added is small, the adhered MDI become thinner layer relatively and contact more effectively with water Consequently it is expected that the adhesion power of stirrer bar to glass vessel becomes lower and the reproducibility of kinetic data is raised. On the basis of these idea, in this report the experiment was conducted on the test solution of as small volume as possible in order to make adhered MDI as thin as possible. And the effect of addition of glass beads in test solution, which increases adhesion site of MDI, on the diasppearance rate of MDI and on thereproducibility of the disappearance was examined.

2-1 Experiments

(1) Materials

MDI and MDA were supplied from Sumitomo Bayer Urethane Co.,Ltd. and Nippon Polyurethane Industry Co.,Ltd., respectively. The properties of these samples are shown below according to the appended informations.

Properties of MDI

Supplier : Sumitomo Bayer Urethane Co.,Ltd.

Purity :99.9% as MDI monomer

Composition :2.4′ -MDI 52.0%

4.4′ -MDI 45.9%

2.2′ -MDI 2.1%

Properties of MDA

Supplier :Nippon Polyurethane Industry Co.,Ltd.

Commercial name :MDA-F

Purity :99.8%

Composition :4.4′ -MDA

Freezing point :91.2 ℃

Water was the deionized and distilled water purchased from Takasugi Seiyaku Co., Ltd..

NIKKOL HCO-20 (Polyoxyethylene hydrogenated caster oil derivatives) and olive oil were purchased from NIKKO CHEMICAL CO.,LTD and Wako Pure Chemical Industries Ltd., respectively.

All other reagents were of guaranteed grade.

(2) Preparation of the dispersed solution of MDI

A hundred milligram of MDI was added to 5 g of HCO-20 or olive oil at 40-50℃ and the mixture was dispersed in water.

(3) Vigorous stirring experiment based on the analysis of individually prepared solutions.

20 ml of water was added in 50 ml erlenmeyer flask with and without about 2g of glass beads of about 2.5 mm diameter and 20 μl of MDI was put on the bottom of the flask. The solutions were stirred at speed of about 3000 rpm. After 0, 2, 5, 15 and 63 hours of stirring at room temperature (about 25℃), 20 ml of toluene solution containing 0.5% dibutylamine were added and stirred for 5 minuts. Tolu-

ene layer was separated from water one by standing and 100 μ l of toluene layer was taken out and diluted with acetonitrile, followed by analysis with HPLC. (detailed anlytical conditions is shown in paragraph 3)

The amounts of residual MDI in the presence and absence of glass beads were compared each other and the effect of the addition of glass beads on decomposition of MDI was examined.

Although purpose of present investigation is to establish the experimental method for vigorous stirring experiment of MDI, for reference the detection of reaction products observed on the way of this experiment were attemped as follows.

After 2 and 63 hours of stirring, aliquots of test solution were centrifuged at 3000 rpm and dissolved organic carbon (DOC) were measured with Shimadzu Total Organic Carbon Analyzer TOC-500 (furnance temperature: 680℃, carrier gas :air, 150 ml/min and injection volume: 10 μ l).

Furthermore toluene extract after 63 hours was injected on ODS column with acetonitrile eluent and on GPC column in order to detect possible reaction product.

2-2 Results

(1) Preparation of dispersed solution using dispersing agent

When MDI was added to HCO-20, violet foaming was observed.

When MDI was added to olive oil, no significant change was observed, but the solution was clouded gradually.

From these results it could be concluded that MDI reacts with these dispersing agents and accordingly further investigation was not conducted.

(2) Vigorous stirring experiment based on the analysis of individually prepared solutions

When the test solution was stirred with glass beads, the solution became turbid by suspending the substance after several hours and white powder appeared on the

surface of the solution after one day.

On the other hand, when glass beads were not added in test solution, most of MDI was adhered on Teflon stirrer bar and the solution was transparent until 2 days at which the solution began to become turbid. Suspended substance was fine in the presence of glass beads than in the absence of these.

Table 1 shows the comparison of the residual MDI with and without glass beads in test solution. Under both conditions, the extremely scattered data were not observed. The amounts of the residual MDI were less in the presence than in the absence of glass beads in all points, which indicate that the addition of glass beads accelerate the decomposition rate of MDI probably as a result of the enlargement of the adhesion area of MDI, which enlarge the contact area of MDI with water, and of stripping off the reaction product from the adhesion sites by collision with glass beads. The difference of the decomposition rate between two conditions correspond to about two times half-life period.

In the present experiment, the adhesion of MDI did not influence greatly the kinetics of MDI decomposition because the reproducibilities of kinetic data in the presence and absence of glass beads were not differ too greatly and the stop of stirring was not observed. Since the main distinction of the present experiment from the preliminary one is the scale of operation, it is presumed that under the condition used in this study MDI is adhered so thin as not to stop the stirring and not to cause poor reproducibility of the data.

(3) Detection of reaction product

Table 2 shows DOC values of the test solution at 2 and 63 hours after stirring under both conditions. DOC values were slightly larger in the presence of glass beads than in the absence of these, but there was not the tendency of increase of TOC values with the progress of reaction.

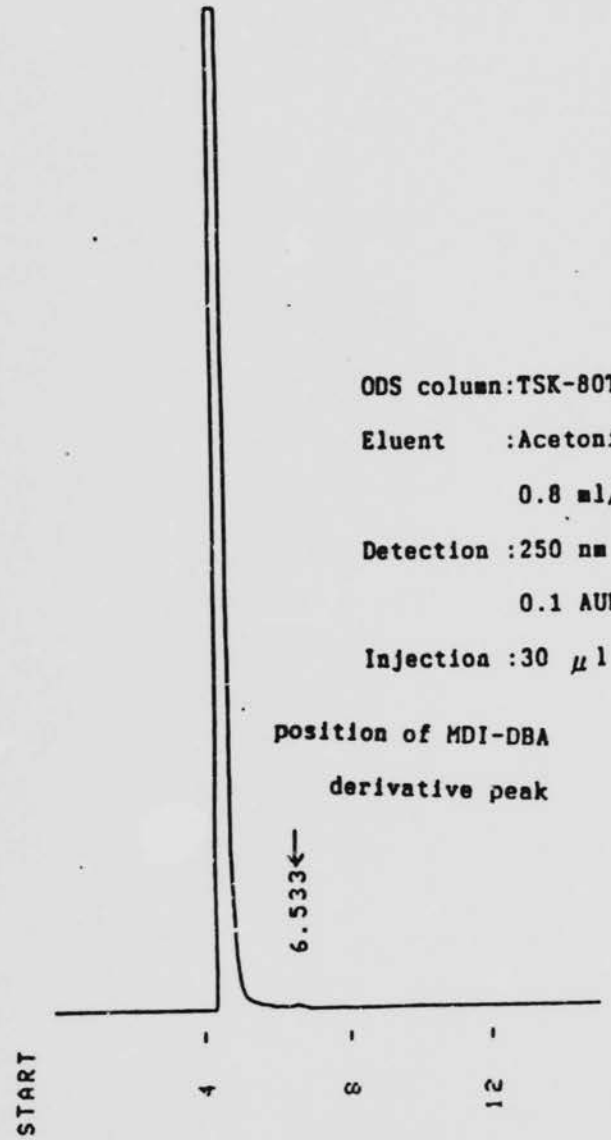
Table 1 Comparison of percent residue of MDIs in vigorous stirring experiments in the presence and absence of glass beads.

Duration of stirring (hours)	In the presence of glass beads		In the absence of glass beads	
	4,4′ -MDI	2,4′ -MDI	4,4′ -MDI	2,4′ -MDI
0	100	100	100	100
2	85.3	88.2	92.5	94.3
5	61.2	68.0	72.6	78.5
15	23.4	33.0	54.3	62.1
63	0	0	5.3	8.3

Table 2 Comparison of DOC values of test solution in vigorous stirring experiments in the presence and absence of glass beads

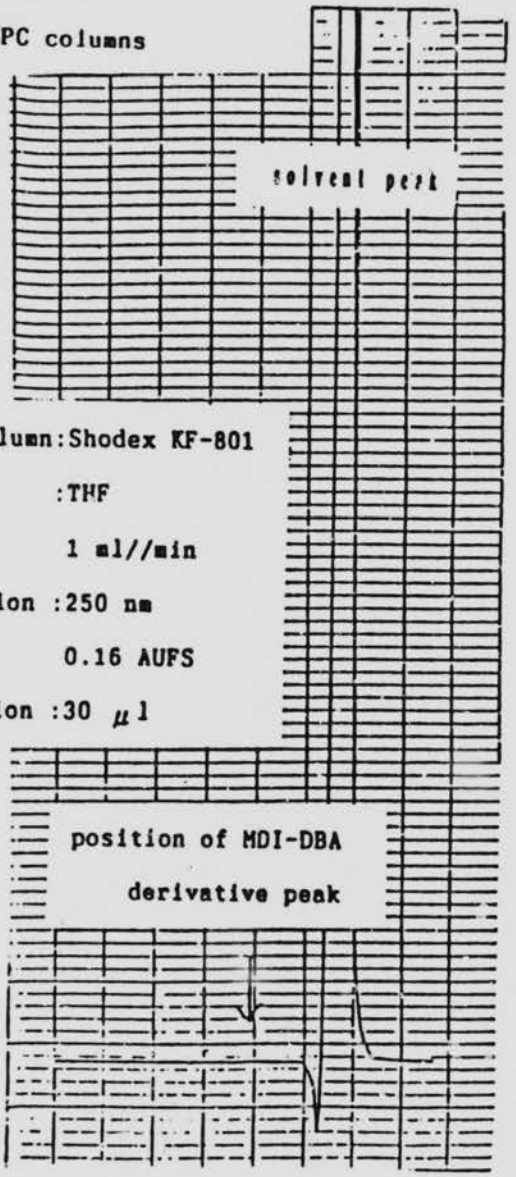
Duration of stirring (hours)	In the presence of glass beads (mgC/l)	In the absence of glass beads (mgC/l)
2	9.83, 10.24	3.22
63	8.12, 9.24	4.60, 6.74

Figure 1 Detection of toluene-soluble reaction product after 63 hours of stirring on ODS and GPC columns



ODS column:TSK-80T
Eluent :Acetonitrile
0.8 ml/min
Detection :250 nm
0.1 AUFS
Injection :30 μl

position of MDI-DBA derivative peak



GPC column:Shodex KF-801
Eluent :THF
1 ml//min
Detection :250 nm
0.16 AUFS
Injection :30 μl

This table also indicates that water-soluble reaction product was less than 10 mg/l as DOC during the period of the disappearance of MDI when MDI was added with the nominal concentration of 1000 mg/l.

Detection of toluene-soluble reaction product was tried on ODS and GPC columns, but any peaks corresponding to reaction products were not observed under the conditions examined (Figure 1).

These results suggest that the majority of MDI were converted in water to polyurea which is not soluble in water and toluene.

3. Study on analytical method of MDI in water

Two methods were examined as the analytical method of MDI in water which is not influenced with the existence of MDA:one is the selective extraction of MDI with a suitable solvent. n-Hexane was examined as such a solvent, but complete separation of MDI from MDA was impossible.

Another method is simultaneous extraction and deactivation of MDI by using the suitable deactivation agent of isocyanate. The agent is required to be highly reactive with isocyanate, to be partitioned to extraction solvent from water and not to interfere with succeeding analysis by HPLC. It is also desirable that MDI isomers are separated by HPLC.

Several aliphatic amines were examined. Primary amines containig pyridyl piperidine formed toluene-insoluble derivatives. Reaction products of MDI with secondary amines were toluene-soluble, but the separation of secondary amine derivatves of MDI isomers by HPLC was dependent on the structure of secondary amine and the composition of the eluent used. Dibutyl amine (DBA) satisfied the necessary conditions as the derivative agent of MDI and the detailed method was investigated to analyze MDI in water by coupling with DBA.

3-1 Experiments

(1) Materials

The same chemicals as in 2-1 (1) were used.

(2) Reaction condition of the coupling with DBA: reaction time and amount of DBA added.

20 μ l of DBA was added to 10 ml of toluene solution of MDI of 1000 mg/l. After 1,15,30 and 50 minutes, 100 μ l of the solution was taken out, diluted 100-fold with acetonitrile and analyzed with HPLC.

After 5,10,15,20 and 40 μ l of DBA were mixed with 10 ml of toluene solution of MDI of 1000 mg/l and stood for about 30 minutes, 100 μ l of the solution was taken out, diluted 100-fold with acetonitrile and analyzed with HPLC.

(3) Analytical method of MDI in water

20 μ l of MDI was added with microsyringe on 20 ml of deionized water in 50 ml erlenmyer flask and mixed with 20 ml of toluene containing 50,100,200 and 400 μ l of DBA for about 5 minutes with magnetic stirrer. After separating toluene layer from water one by standing, 100 μ l of toluene solution was taken out, diluted with acetonitrile and analyzed with HPLC.

The same operation was carried out with chloroform instead of toluene in order to find suitable extraction solvent.

(4) Influence of MDA on analysis of MDI in water

10 μ l of MDI was added with microsyringe to 20 ml of deionized water in 50 ml erlenmyer flask containing 0.2 ml of 2 % solution of MDA in acetnitrile and it was immediately mixed with 20 ml of 0.5 % toluene solution of DBA for about 5 minutes with magnetic stirrer. After separating toluene layer from water by standing, 100 μ l of toluene solution was taken out, diluted with acetonitrile and analyzed with HPLC.

The standard solution for the determination of the recovery of the extraction

was prepared by the same procedure as 3-1(1) and the recovery of MDI from water was calculated by dividing the peak area of DBA derivative of MDI measured in 3-1 (3) or (4) by one of the standard solution.

(5) HPLC condition

DBA derivative of MDI was determined under the following condition using ODS column.

Column :TSK 80-T (4.6 mmI.D. x 250 mmL)

Eluent :Acetonitrile/water=15/85(v/v)

0.8 ml/min

Detection:HITACHI L-4000 UV-Detector

250 nm,0.1 AUFS

Injection:30 μ l

Record :Shimadzu Chromatopak C-R4A

3-2 Results

Typical chromatogram of the reaction product of MDI with DBA is shown in Figure 2. Two peaks were detected under the condition used,in which the first peak with small retention time corresponds to 4,4' -isomer of MDI.

(1) Reaction condition of the coupling with DBA

Table 3 shows the variation of the peak areas with reaction time. Both peak were constant in the range of 1 minute to 50 minutes of the reaction times indicates that the coupling reaction with DBA is completed within 1 minute.

Table 4 shows the variation of peak areas with the amount of DBA added. Peak areas increased with increase of DBA added and reached to constant value above 15 μ l of DBA added. When the amount of DBA added was less than 10 μ l, other two peaks with smaller retention times were observed, which are presumed to be MDI

itself and the reaction product half coupled with DBA.

Molar ratio of DBA to MDI is 2.2 (= $8.8X10^{-5}/4.1x\ 10^{-5}$ moles) for the addition of 15 μl of DBA. Consequently the reaction was conducted quantatively.

Figure 3 shows the calibration curve when 10 ml of MDI toluene solutions ranging in concentration from 50 to 1000 mg/l were reacted with 20 μl of DBA. Good linear relationship between the concentration and the peak area was observed.

(2) Analytical method of MDI in water

Table 5 shows the variation of the recoveries of MDI from water with the amount of DBA added when toluene was used as the extraction solvent. Recoveries were constant within the experimental error for the addition above 50 μl of DBA and the average recoveries were 95.9 and 97.9 % for peak 1 and 2,respectively.

When chloroform was used as a extraction solvent, the recoveries were dependent on the amount of DBA added. Consequently chloroform is not suitable as the extraction solvent.

(3) Influence of MDA on analysis of MDI in water

Table 6 shows the recoveries of MDI in the presence of MDA. Measurement were carried out in triplicate and the average recoveries for both peaks were 92.5 and 96.1 %,respectively, which were consistent with the values obtained in the absence of MDA within the experimental error. It is therfore concluded that the method established in this study is not influenced by existence of MDA.

(4) Recommended condition of the analysis of MDI using DBA.

① Concentration of DBA

Since the reaction of MDI with DBA proceeded almost quantitatively,the standard solution of DBA derivative of MDI can be prepared by reacting with toluene solution containing DBA equivalent to MDI analyzed. On the analysis of MDI in water a part of DBA in toluene is partitioned in water and hence the amount of DBA

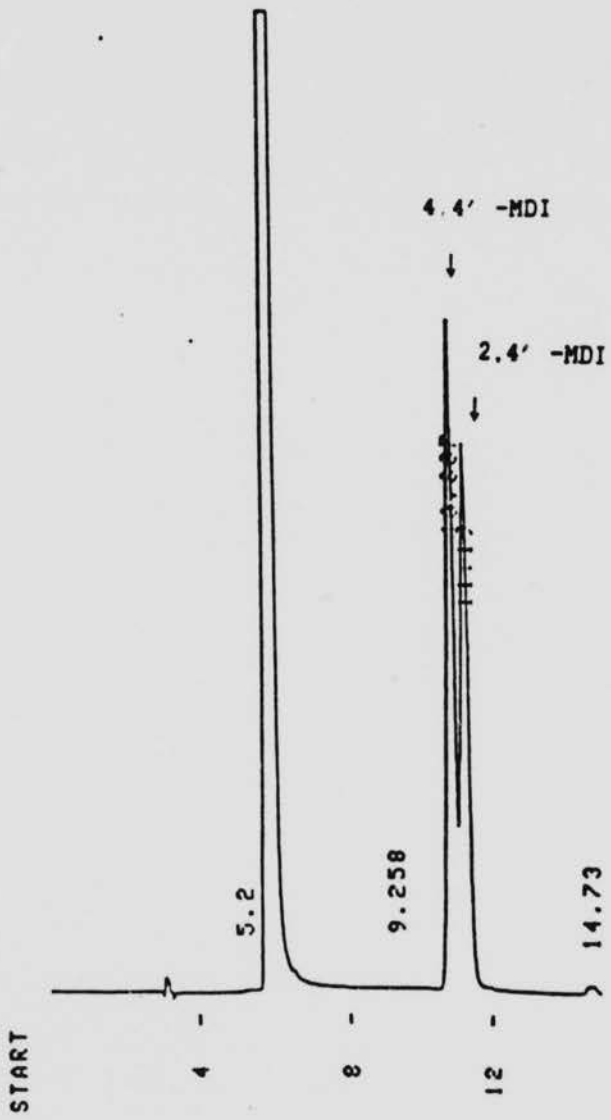


Figure 2 Typical chromatogram of MDI derivative by DBA

Column :TSK-80T

Eluent :Water/Acetonitril-15/85(v/v)

0.8 ml/min

Detection:250 nm

0.1 AUFS

Injection:30 μl

Final concentration of the sample was 10 mg/l as MDI

Table 3 Variation of peak area for MDI derivatives by DBA with reaction time

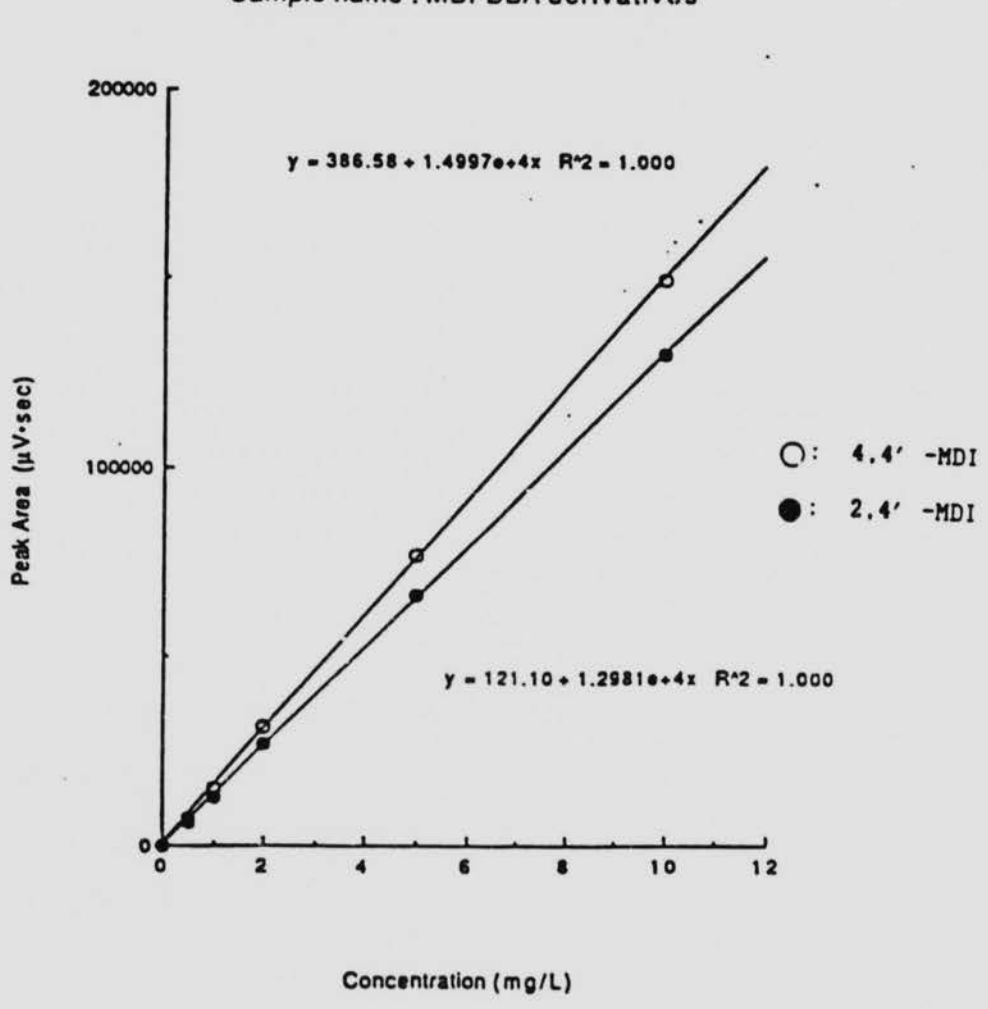
Reaction Time (min)	Peak area (μV · sec)	
	4,4′ -MDI	2,4′ -MDI
1	154152	129538
15	151170	131723
30	156253	132615
50	153222	131662
100	150655	131235

10 ml of MDI toluene solution of 1000 mg/l was reacted with 20 μl of DBA.

Table 4 Variation of peak area for MDI derivatives by DBA with the amount of DBA added

Amount of DBA (μl)	Peak area (μV · sec)	
	4,4′ -MDI	2,4′ -MDI
0	0	0
5	34375	21819
10	100951	64750
15	161214	137075
20	156253	132615
40	154793	133383

10 ml of MDI toluene solution of 1000 mg/l was reacted with various amounts of DBA for about 30 min.



Fifure 3 Calibration curve of HPLC analysis for MDI derivative by DBA.
Analytical condition is the same as Figure 2.
The number on the absissa indicates the final concentration of MDI derivative as MDI.

Table 5 Variation of recovery of MDI from water with the amount of DBA added

Amount of DBA (μl)	Recovery (%)	
	4,4' -MDI	2,4' -MDI
50	96.92	99.79
100	97.68	98.27
150	98.62	99.67
200	92.88	96.15
400	93.41	95.81

20 μl of MDI in 20 ml of water was extracted with toluene solution containing various amounts of DBA.

Table 6 Influuence of MDA on recovery of MDI from water

Repetition No.	Recovery (%)	
	4.4' -MDI	2.4' -MDI
1	95.27	98.03
2	91.12	95.16
3	91.10	94.64
Average	92.50	94.64

10 μl of MDI in 20 ml of water containing 4 mg of MDA was extracted with toluene solution containing various amounts of DBA.

necessary to derive entire MDI is more than one necessary on the preparation of standard solution. From Table 5, when MDI in water is extracted bythe equivalent volume of toluene solution of DBA, the amount of DBA above 2 timesof MDI analyzed on the basis of mole is adequate to achieve the recovery above 95 percent.

② Reaction time

The reaction of MDI with DBA proceeded instantaneously in both the preparation of standard solution and the extraction from water. Consequently it is not neccesary to take the reaction time into account.

③ Extraction solvent

Toluene is better than chloroform.

4. Discussion

(1) Method for vigorous stirring experiment of MDI

In the present investigation two methods were examined as the method for vigorous stirring experiment of MDI, but the method using the dispersed solution is not applicable to MDI because MDI reacted with dispersing agent. On the other hand, in the method based on the analysis of individually prepared solution at each time of the disappearance of MDI, the experiment is able to be conducted without the stop of stirring and lack of the reproducibility of kinetic data if the volume of test solution is made as small as in present investigation.

Although the addition of glass beads accelerated the disappearance rate of MDI probably because of the enlargement of adhesion site of MDI, the method without the addition of glass beads is recommended as the method for vigorous stirring experiment of MDI because it more resemble the naturally occuring process.

(2) Analytical method of MDI

MDI was analyzed by coupling with DBA, followed by quantification by HPLC. The coupling reaction proceeds almost quantitatively and instantaneously and MDI isomers were separated on ODS column.

This method have the advantages over one using pyridyl piperidine as a coupling agent in following point: One is that DBA derivative of MDI is toluene-soluble, which makes the simultaneous operation of the extraction from water and the coupling with DBA possible by using the toluene solution of DBA, while the reaction product with pyridyl piperidine was less soluble to toluene.

Another advantage is that DBA derivrtive of MDI have less adsorptive nature to ODS gel than pyridyl piperidine derivative, which makes the maintenance of HPLC column easy.

(3) The fate of MDI in water

In this investigation, the disappearance rate of MDI in water and the reaction products were evaluated preliminarily though these results are only a rough estimation. Half-lives of MDI in water were several times longer compared with TDI.

The water-soluble reaction product less than 10 mg/l as DOC was found, but its constituents were not identified in the present study.

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